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Volume 9
Number 97 *Method of determining the moisture content of butter.*
Article 1

August 2017

Method of determining the moisture content of butter.

G. L. McKay
Iowa State College

John Bower
Iowa State College

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McKay, G. L. and Bower, John (2017) "Method of determining the moisture content of butter.," *Bulletin*: Vol. 9 : No. 97 , Article 1.
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BULLETIN 97 JUNE, 1908

EXPERIMENT STATION

IOWA STATE COLLEGE
OF AGRICULTURE AND MECHANIC ARTS

DAIRY SECTION

METHODS OF DETERMINING THE MOISTURE CONTENT OF BUTTER

AMES, IOWA

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METHOD OF DETERMINING THE MOISTURE CONTENT OF BUTTER.

G. L. McKAY JOHN BOWER

Introduction

The commercial value to those engaged in creamery management of knowing the moisture content of butter has created a want for some simple and accurate means of ascertaining the percentage of water in butter. The recent stringent enforcement of the pure food law has emphasized its need in the case of dealers. To meet the demand several methods have been devised and placed upon the market. In the use of these, reliability of results has been questioned, and it has been felt that official tests should be made to determine their accuracy.

This bulletin gives a description of the methods commonly used, with comments upon them. Their results are compared with the standard gravimetric analysis recognized by the Association of Official Agricultural Chemists. In making comments, the writers have taken into consideration conditions as they exist throughout the creameries. Simplicity of method, cost of apparatus, expense of manipulation and the intelligence of the labor employed, are also dealt with. Variations in the water content, so far as they affect the results of the methods, are given general consideration.

A full description of a method of moisture determination lately devised and now used by this Station is presented for the first time. Notes on the preparation of sample, with tables showing variation in water content found in samples taken from different parts of churn and tub, are also contained in this bulletin.

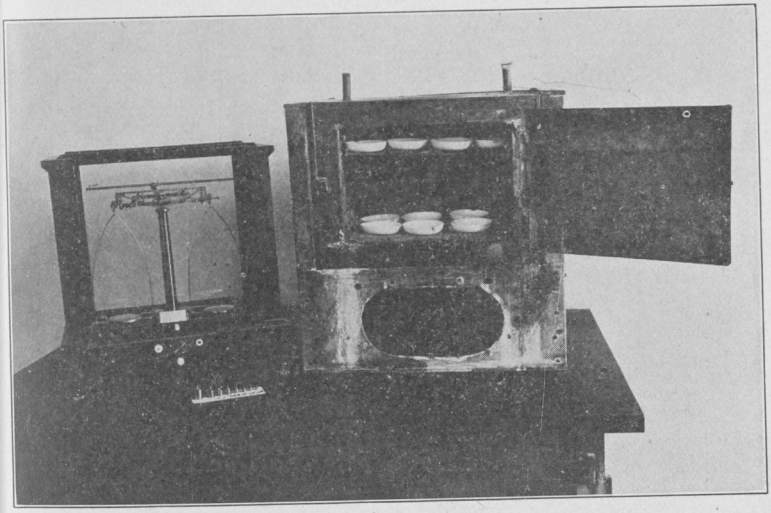
At present the methods may be divided into those which require a chemical balance, those in which more simple scales are in general use, and those where the determination is made by application of centrifugal force using specially constructed glassware. The Official Method, the Richmond, and the Geldard Butter Tester may be classified in the first class; the Patrick, Irish, Gray and Wisconsin High Pressure Oven belong to the second class; the Wagner Butter Hygrometer, and special Babcock butter bottles belong to the third class.

The comparisons given in the tables represent the work of J. C. Brown, J. Bower and W. G. McKay. Unless otherwise credited, they are the results obtained by Mr. Brown.

GRAVIMETRIC ANALYSIS OR OFFICIAL METHOD.

In the gravimetric analysis the apparatus required is as follows: Chemical balance, evaporating dishes having a surface of 20 square centimeters, a large dessicator, and one drying oven with surrounding jacket to contain water. The water

is heated by gas, alcohol, kerosene or gasoline. The temperature of the oven is maintained by keeping the water slowly boiling. This gives a uniform temperature in oven of 212 degrees F. In cities or towns where gas is available a single gas jet is sufficient to maintain the required temperature. An



Apparatus Used in the Official Method.

oven without outside water jacket may be used. Such an oven requires more attention, and temperature control is more difficult.

From 1.5 to 2.5 grams of the sample are dried to a constant weight in dishes, previously dried and cooled. The drying process requires from five to six hours.

The objections that are generally raised to this method are: first, the length of time required; second, the cost of apparatus; third, the unusual degree of precision required; fourth, the necessity of suitable place for operation. To the first of these objections it has been the writer's experience that in the analysis of butter the gravimetric method as described above is by far the quickest of all methods. By this is meant the actual time required by the operator in analyzing any number of samples is less than any other method. Among those not informed, it is thought that it requires from five to six hours to make a moisture determination. Only a few minutes is required to do the actual work. The rest of the time is consumed in drying the butter. An operator can readily weigh out from 12 to 24 samples per hour. If porcelain or aluminum dishes are used they may be burned to a constant

weight and weight recorded. If they are cleaned thoroughly after each determination the necessity of reweighing the dish would thus be obviated and time saved. They should, however, be weighed from time to time to insure constant weight. Some can weigh as many as 50 samples in an hour. It would only be required to reweigh at the end of the drying period and calculate percentage. This could be done as follows:

Weight of dish and sample minus weight of dish equals weight of sample used.

Weight of dish and sample minus weight of dish and sample after being subject to heat equals weight of water evaporated.

Weight of evaporated water divided by weight of sample multiplied by 100 equals per cent moisture.

Example.

Let 28.3567 gr. equal wt. of dish.
Let 29.9367 gr. equal wt. of dish and sample.
Then 29.9367 minus 28.3567 equals 1.58 gr., weight of sample.
After drying the dish and sample weight is now 29.7342.
Then 29.9367 minus 29.7342 equals .2025 gr. or water evaporated.

$$\text{Then } \frac{.2025 \text{ times } 100}{1.58} \text{ equals } 12.81\%.$$

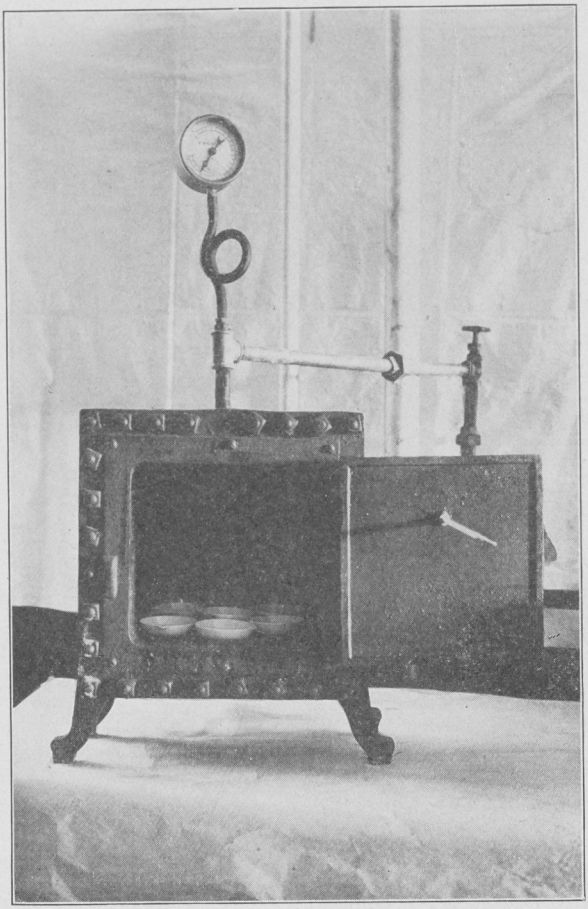
The cost of apparatus and necessity of providing a suitable place to keep chemical balance would perhaps be the chief objection. The cost need not exceed $60. When it is considered that some creameries are losing this sum in a few weeks, and in some places a few days, the force of the objection is lost. Many creameries have a separate testing room where the balance may be kept in good condition. Such a room can readily be provided.

In regard to degree of precision required the gravimetric method is not beyond the average buttermaker. The students of the one year dairy course at Ames, after a little practice, were without exception able to make a moisture determination very accurately. Buttermakers, who are capable of a careful manipulation of the Babcock test, can be trusted to give satisfaction in making a moisture determination. The absolute certainty of this method, as compared with some others would more than make up for the extra cost involved.

In the larger creameries the chemical balance could be used in making a complete analysis of butter. This is sometimes very desirable and necessary if managers are to be in a position to control the working process upon a satisfactory basis. If a butter fat as well as a moisture standard be adopted the analysis for butter fat would be required to better maintain composition of butter within the standard allowed by law.

WISCONSIN HIGH PRESSURE OVEN.

This oven is a product of investigations at the Wisconsin Station. It is made of cast iron, and in size is about 12 inches square, with an inner shell about 9 inches square. A space for circulation of steam is left between the outside and inside castings of the oven. It is of sufficient strength to resist steam pressure coming directly from the boiler. From 60 to 80



THE WISCONSIN HIGH PRESSURE OVEN

pounds pressure is used, obtaining a temperature of 240 to 280 degrees F. This will vary with the distance of the oven from the boiler and the steam pressure there.

In weighing the sample a Torsion balance No. 1900 was used at the Wisconsin Station. This scale is sensitive to one one-hundredth of a gram and may be used by a buttermaker.

About ten grams of the butter to be tested are weighed into a 3-inch, flat bottom aluminum dish having sides three-fourths of an inch high. If exactly ten grams are taken no record need be made of the weight. The dish is then placed in the high pressure oven which has previously been heated up to a temperature above that of boiling water by opening the valve which allows steam to enter the steam chamber of the oven. The sample of butter is heated in this oven for half an hour, then taken out, allowed to cool, is weighed and recorded. The sample is then placed in the oven another half hour in order to be sure of removing all the water.

It is also necessary when taking the aluminum dishes out of the oven to place them in some perfectly dry, well protected place, and weigh them as soon as cool, as the dish and the butter will take up moisture if allowed to stand around too long before weighing.

This oven may be obtained for $20 and the Torsion balance for $10. Aluminum dishes may be obtained for 25c each.

Under the head of special precautions, the following is recommended by the Wisconsin Station in the use of this oven:*

"1. The weighing scale must be easily sensitive to .05 grams if 50 grams of butter are tested; and to .01 grams if 10 grams are taken.

"2. The scales must be properly adjusted, kept in a clean dry place, and protected from drafts of air while in use.

"3. The drying pans should be from 4 to 5 inches in diameter when 50 grams of butter are tested.

"4. The clean, empty drying pans should be heated just before weighing in order to completely dry them.

"5. The butter should be heated until it reaches a constant weight, a second heating and weighing being always recommended.

"6. The hot pans should be placed on a clean piece of tin or a porcelain plate, when taken from the oven to cool.

"7. Never weigh the pans while hot, nor after standing an hour or more outside the oven, as they may take up moisture from the air."

To calculate the percentage of water in a 50 gram sample, multiply the loss of weight by 2. If the loss is 7.5 grams the moisture content is 15%. If a 10 gram sample is used, multiply the loss by 10 and the result will be the percentage water content.

The results obtained in the use of the Wisconsin High Pressure Oven were at first unsatisfactory. The oven sent to this Station was found to be imperfectly constructed. The steam penetrated to the inside of the oven in the form of small particles of water which immediately evaporated. Instead of the air being dry enough to absorb the water as it was driven off from the butter samples, it was in part saturated with the moisture from escaping steam and as a result it was impossible to determine the moisture content accurately. Results were invariably too low. The oven was rejected and a second one

*Bulletin 154, University of Wisconsin, Agr. Exp. Sta.

procured which proved to be correctly constructed. It is essential, therefore, that such an oven, if used, should be so constructed that there is no danger of steam penetrating through the inner plate. It should be tested before shipment.

Comparisons of Results Obtained by the Official and Wisconsin Tests.

No.	Official Method	Wisconsin Method	Difference	No.	Official Method	Wisconsin Method	Difference
1	15.24	15.00	—.24	2	14.27	14.21	—.06
3	16.53	16.45	—.08	4	16.03	16.27	.24
5	16.42	16.60	.18	6	13.57	13.52	—.05
7	14.34	14.50	.16	8	13.44	13.39	—.05
9	15.98	15.99	.01	10	14.16	14.51	.35
11	14.30	14.47	.17	12	14.54	14.54	.00
13	14.70	14.68	—.02	14	14.53	14.52	—.01
15	14.60	14.74	.14	16	14.72	14.58	—.14
17	15.59	15.40	—.19	18	12.84	12.80	—.04
19	12.66	12.93	.27	20	12.99	13.32	.33
21	15.85	15.62	—.23	22	15.54	15.64	.10
23	15.97	16.05	.08	24	15.46	15.58	.12
25	16.51	16.50	—.01	26	15.86	16.05	.19
27	16.52	16.35	—.17	28	14.79	15.00	.21
29	14.86	14.81	—.05	30	12.55	12.52	—.03
31	13.20	13.07	—.13	32	16.84	17.00	.16
33	16.32	16.08	—.24	34	15.79	15.83	.04
35	16.09	16.27	.18	36	16.60	16.60	.00
37	17.60	17.45	—.15	38	17.60	17.45	—.15
39	15.22	15.41	.19	40	14.16	14.23	.07
41	15.26	15.04	—.22	42	15.64	15.72	.08
43	15.68	15.50	—.18	44	14.71	14.55	—.16
45	15.06	15.25	.19	46	15.68	15.67	—.01
47	15.71	15.75	.04	48	15.75	15.73	—.02
49	13.72	13.94	.22	50	14.00	13.85	—.15
51	14.62	13.72	.10	52	15.47	15.40	—.07
53	16.36	16.39	.03	54	15.06	14.85	—.21
1	11.81	11.85	.04	2	12.4	12.2	—.20
3	15.20	15.1	—.10	4	15.41	15.15	—.26
5	15.48	15.25	—.23	6	15.75	15.8	.05
7	12.95	13.1	.15	8	12.37	12.3	—.07
9	15.38	15.1	—.28	10	14.30	14.25	—.05
11	14.99	14.75	—.24	12	14.19	14.1	—.09
13	14.16	14.25	.09	14	14.17	14.0	—.17
15	14.12	14.2	.08	16	15.07	15.39	—.32
17	14.87	14.7	—.17	18	14.60	14.65	.05
19	15.72	15.88	.16	20	16.01	16.36	.35
21	15.68	15.70	.02	22	14.60	14.55	—.05
23	14.65	14.85	.20	24	16.65	16.6	—.05
25	16.45	16.6	.15	26	16.18	16.5	.32
27	14.15	14.0	—.15	28	15.87	15.95	.08
29	13.36	13.5	.14	30	13.45	13.3	—.15
31	12.42	12.0	—.42	32	13.49	13.6	.11
33	10.79	10.65	—.14	34	15.10	14.8	—.30
35	17.52	17.2	—.32	36	15.83	15.5	—.33
37	15.22	15.0	—.22	38	12.28	12.1	—.18
39	14.70	15.0	.30	40	20.72	20.9	.18
41	20.06	19.75	—.31	42	21.00	21.14	.14
43	13.62	13.55	—.07	44	13.85	14.06	.21
45	13.68	13.60	—.08				

In the first 54 of the samples analyzed a chemical balance was employed. In the remainder a scale made by the Torsion Balance Co., style 1500, No. 26804, was used. Where the balance was employed it was found that 11 samples differed from the Official Method by over .2%; of these 11, two differed by .3%, and none exceeded .4% difference. Where the Torsion scale was used 16 showed an error of over .2%. Of these, eight differed from the Official Method by .3% and one only exceeded the .4% difference and that by a small margin. Quite a number of cases showed a lower percentage than that obtained by the Official Method.

The Wisconsin Method differs from other methods, particularly the Richmond and the Patrick or Irish methods, in that there is better control of temperatures. At between 40 and 60 lbs. pressure a temperature of from 240 to 280 degrees is readily obtainable. This pressure may easily be obtained where boiler pressure is maintained above 70 lbs. Should, however, the pressure fall below 40 lbs. at the oven, results will be low, unless longer time is given to evaporate the water.

The possible use to which such an oven may be put in preparation of mother starters readily appeals to the maker. Since there is no pressure inside the oven, a higher temperature than the boiling point of liquid used can not be obtained.

A low pressure steam oven was also suggested and used by Professor H. H. Dean, of Guelph, Canada. The Wisconsin High Pressure Oven is quite distinct from that recommended by Professor Dean.

Below is found further table of comparison of results obtained by High Pressure Oven and the Official Method.*

PER CENT WATER IN BUTTER.

	Official Method	Wisconsin High Pressure Oven Method
Sample No. 1	13.05 13.20	13.1 13.1
Sample No. 2	18.71 18.92	19.0 19.1
Sample No. 3	20.89 20.90	21.0 21.0
Sample No. 4	12.37 12.25	12.5 12.45
Sample No. 5	18.77 18.59	18.4 18.6

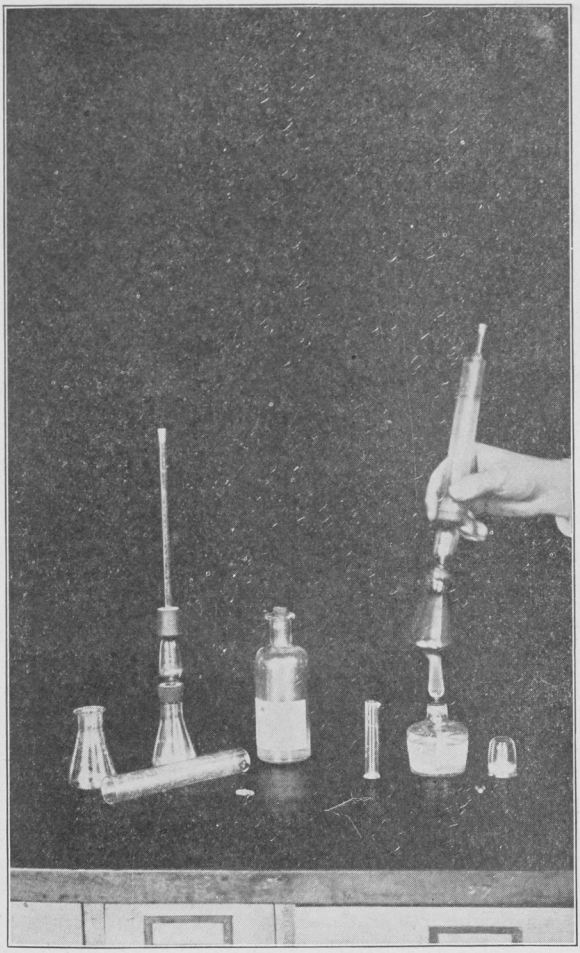
GRAY'S METHOD.

The Gray Method was devised by C. E. Gray, formerly Assistant Dairyman in charge of Butter Investigations, Dairy Division, Bureau of Animal Industry. A full description of it

*Bulletin No. 154, Wisconsin Experiment Station.

is given in circular No. 100, published by the U. S. Department of Agriculture.

According to directions there given, a ten gram sample of butter, carefully prepared, is weighed on a piece of parchment paper. This is placed in a small pear-shaped flask and 6 cc of amyl reagent, consisting of five parts of amyl acetate and one part amyl valeriante, is added. A distilling apparatus is then



Operating the Gray Test.

connected with this flask by means of a rubber cork. Heat is applied and the water in the sample boils and passes as steam into the tube where it is condensed and trapped. Care must

be taken that the steam does not escape through the application of too much heat. Foaming is usually prevented by the use of 6 cc of the reagent though in some samples a trifle more is required.

When the mixture in the flask becomes a brown color, and all the crackling noises cease, it may be concluded that all the water has been driven from the flask. This takes not less than five minutes and with most samples need not be more than eight minutes.

After disconnecting the flask from the stopper, place the glass stopper in the tube, giving it a slight turn to insure its being held firmly. Carefully invert the tube, holding the mouth of a small inner tube upwards and pour water from the condensing jacket. This may then be removed.

To separate the reagent and water and to get the last traces of water down into the graduated part, the tube is held with the bulb in the palm of the hand and the stoppered end away from the body, raised to a horizontal position, and swung at arm's length sharply downward to the side. This is repeated a number of times until the dividing line between the water and amyl reagent is very distinct and no amyl reagent can be seen with the water or vice versa. The tube should then be held a short time with the stoppered end downward and the amyl reagent in the bulb of the tube agitated in order to rinse down any water that may be adhering to the sides of the bulb. The reading should not be taken until the tube and its contents have cooled so that very little warmth is felt. The water is in the bottom of the tube, and when a ten gram sample is taken the percentage may be read directly.

The flask may be cleaned by washing with soap, washing powder, or washing soda in hot water. It is not absolutely necessary to wash it after each determination; the residue may be poured out and the flask wiped with a cloth or thin paper. The flask must always be dry (free from water) before making a determination.

After making the test, empty the tube by holding the stoppered end downward, removing the stopper and allowing the contents to flow out quickly. In this way the amyl reagent runs out after the water and carries with it practically all the water, which might otherwise adhere to the tube. The tube, after emptying, should be swung in the manner described for separating water from amyl reagent, which will almost completely empty it. Following this plan it is not necessary to dry the tubes after each determination. Occasionally they should be washed carefully with a hot solution of sodium carbonate (sal soda) and thoroughly dried before using.

Comparisons of Results Obtained by the Official and Gray's tests.

No.	Official Method	Gray Method	Difference	No.	Official Method	Gray Method	Difference
1	15.90	16.36	.46	2	12.94	14.2	1.26
3	15.45	15.8	.35	4	15.27	15.3	.03
5	14.16	14.23	.07	6	14.90	15.00	.10
7	19.10	19.9	.80	8	16.38	16.8	.42
9	18.27	19.1	.83	10	15.95	15.8	—.15
11	17.32	17.85	.53	12	19.25	19.65	.40
13	14.81	15.2	.39	14	17.68	17.5	—.18
15	16.96	17.4	.44	16	15.50	16.0	.50
17	14.68	15.1	.42	18	13.62	13.8	.18
19	13.38	13.3	—.08	20	14.86	15.35	.49
21	13.80	14.8	1.00	22	12.91	13.1	.19
23	14.02	14.2	.18	24	13.17	13.15	—.02
25	12.94	13.2	.26	26	13.63	13.8	.17
27	11.07	11.4	.33	28	14.20	15.1	.90
29	14.66	14.7	.04	30	15.17	15.1	—.07
31	14.26	15.2	.94	32	12.06	12.3	.24
33	13.88	13.9	.02	34	14.10	13.9	—.20
35	13.64	13.4	—.24	36	14.82	15.0	.18
37	14.08	13.7	—.38	38	13.71	14.2	.49
39	12.41	13.1	.69	40	13.30	13.3	.00
41	11.77	12.2	.43	42	14.60	15.3	.70
43	14.88	15.8	.92	44	12.62	13.3	.68
45	15.24	15.2	—.04	46	14.27	14.9	.63
47	16.53	17.5	.97	48	16.42	17.2	.78
49	16.03	16.8	.77	50	16.42	16.5	.08
51	14.34	15.0	.66	52	13.44	14.0	.56
53	15.98	16.2	.22	54	14.16	15.3	1.14
55	14.30	14.4	.10	56	14.54	14.4	—.14
57	14.70	14.4	—.30				

No.	Official Method	Gray Method	Difference	No.	Official Method	Gray Method	Difference
1	14.04	14.0	—.04	2	13.12	12.7	.58
3	13.24	13.3	.06	4	14.30	14.0	—.30
5	13.69	14.8	1.11	6	12.76	13.4	.64
7	13.00	13.7	.70	8	11.54	10.9	—.64
9	12.84	12.0	—.84	10	12.97	12.0	—.97
11	12.92	12.2	—.72	12	12.43	12.6	.17
13	15.06	15.0	—.06	14	14.53	14.2	—.33
15	14.64	14.1	—.54	16	15.4	15.4	.00

—By W. G. McKAY.

The above results were obtained by using such glassware and reagents as were available in the open market. Directions as to method were at first followed closely. It was found, however, that to get even approximate results required considerably more time in the heating than was recommended. Even with this precaution very variable results were obtained. Results as indicated above show both a higher and a lower mois-

ture content than the Official Method. The latter condition is particularly to be guarded against. Any method which is liable to give a lower moisture content than that actually contained in the sample is not a safe method to use. The butermaker is working under a sense of false security. Low results may be obtained from several causes; first, the sample may not be heated long enough, in this way the water is not all driven off from the butter; second, the rubber stopper may not fit the neck of flask; third, too rapid evaporation, allowing escape of evaporated water, and fourth, by failing to get all the condensed water into the graduated portion of the apparatus. Another cause would be incorrect calibration of the glass ware. This would also be responsible for too high a reading. If complete separation of reagent from water is not made, using centrifugal force as recommended, the reagent increases the volume of water to be measured. To one or all of these causes may be attributed lack of uniformity of results given above. Incorrect calibration of glassware and particularly the impurity of reagent were the main factors influencing unfavorably the above results.

Through Mr. Gray the writers were able to obtain pure reagents and glassware constructed according to directions.

The following table gives the results obtained:

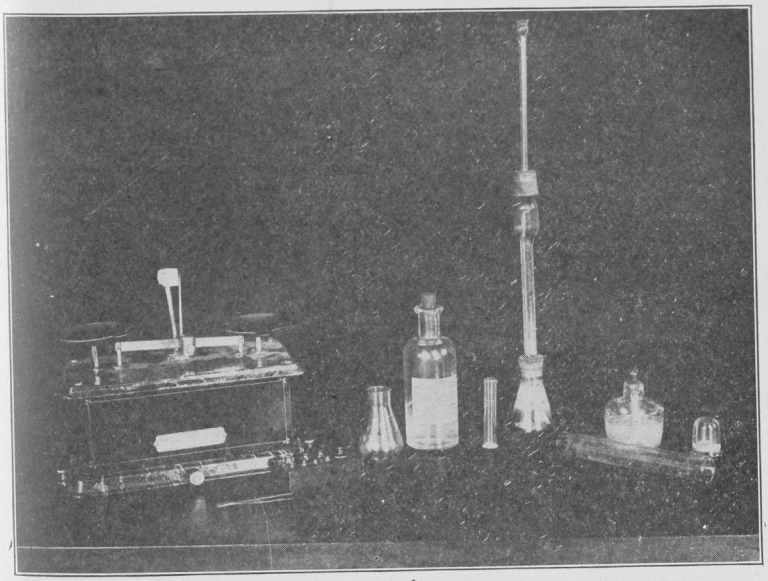
No.	Official Method	Gray Method	Difference	No.	Official Method	Gray Method	Difference
1	12.48	12.2	—.28	2	10.92	10.85	—.07
3	12.04	12.25	.21	4	11.01	11.15	.14
5	13.99	14.10	.11	6	14.72	14.8	.08
7	13.12	13.25	.13	8	14.71	14.85	.14
9	17.38	17.2	—.18	10	15.21	15.3	.09
11	14.04	13.8	—.24	12	15.90	16.20	—.30
13	15.34	15.35	.01	14	13.10	13.4	.30
15	14.22	14.4	.18	16	12.30	12.35	.05
17	16.09	16.30	.21	18	15.19	15.3	.11
19	17.16	17.00	—.16	20	15.08	15.00	—.08

The above table is an indication of what may be done by this method where apparatus is constructed according to directions, where the purity of reagent is unquestionably correct and where operation is performed according to directions. The errors found in the first table were errors not due to method but to other factors already indicated. They are presented not in condemnation of the method but rather to show how much dependence may be placed upon such apparatus and reagents as may be obtained on the market. There are many makers who might well question results obtained by this method. Much of the earlier apparatus was not constructed according to recommendations of the inventor. It was im-

properly calibrated; the glass stopper was ill fitting, allowing escape of water in shaking; the heating flask was so constructed in its neck that the rubber cork did not fit snugly, allowing water to be driven and held between the cork and the neck of the bottle.

The fragile glassware and cost of reagent are factors that would have to be considered. It has the advantage that it does not require a costly scale or balance and it may be operated under conditions that would affect unfavorably other methods.

With the introduction of the Gray test came modifications of the method. The Wagner Improved Method differed from the method devised by Gray in the construction of apparatus. The condensed steam was collected directly in the graduated portion of the apparatus. By this means, it was claimed, there



THE WAGNER APPARATUS.

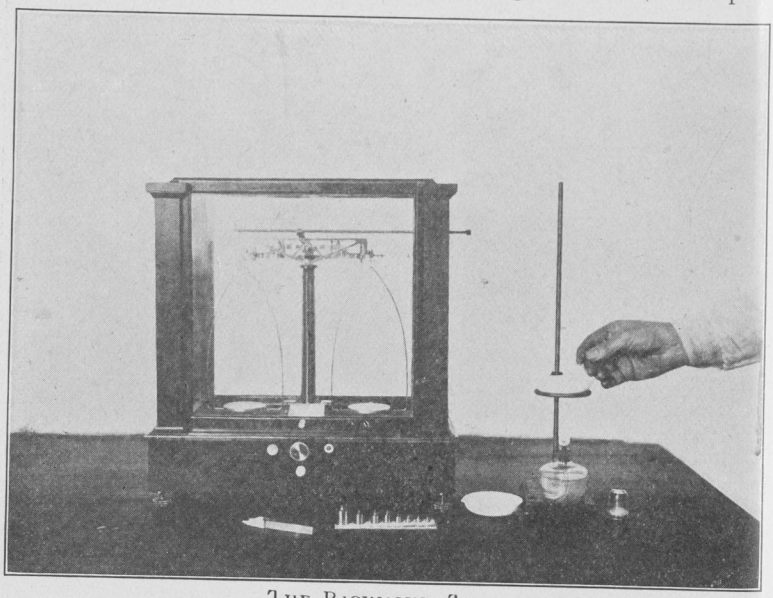
was no necessity of shaking the apparatus to get separation of water from reagent. This was found to be incorrect. The difficulty of freeing graduated portion from moisture after each determination made this method more difficult to operate and more uncertain in results.

Below are given results obtained by W. G. McKay with apparatus and reagent supplied by a firm dealing in dairy supplies.

No.	Official Method	Wagner Method	Difference	No.	Official Method	Wagner Method	Difference
1	13.12	13.0	—.12	2	14.88	14.90	.02
3	12.74	12.75	.01	4	14.97	15.65	.68
5	13.60	14.25	.65	6	15.84	16.40	.56
7	13.52	12.70	—.82	8	14.95	14.5	—.40
9	14.02	13.9	—.12	10	15.53	15.6	.07
11	14.15	14.3	.15	12	12.83	13.6	.77
13	12.28	13.0	.72	14	14.27	14.2	—.07
15	11.74	12.8	1.06	16	11.48	13.7	2.22
17	14.69	15.6	.91	18	11.39	12.2	.81
19	14.92	16.2	1.28	20	11.75	12.6	.85
21	14.30	15.5	1.20	22	12.26	13.3	1.04

RICHMOND METHOD.*

About ten grammes are weighed out into a small porcelain basin provided with a glass stirrer. This is placed over a very small flame, or on a sand-bath, and the butter carefully, but vigorously, stirred till all signs of frothing cease. The temper-



THE RICHMOND TEST.

ature must be so regulated that sputtering is avoided, and that the "curd" does not become browned by the heat. The basin

*Page 252, Dairy Chemistry, Henry Droop Richmond.

and its contents are, after cooling, weighed; the loss of weight indicates water.

In this method which is the most rapid of all the methods a chemical balance was used. Care was taken to avoid sputtering by vigorous stirring while at the same time removing the basin from the flame when there was danger of losing weight through this cause. Directions in this respect were followed closely. Results obtained by using an aluminum basin did not show the regularity in results that were obtained when the porcelain vessel was used.

From two and a half minutes to three minutes are all that are required to complete the evaporation of the moisture contained in the sample. One can not work according to time, but must follow the directions as given above. In ten to twelve minutes a complete determination may be made.

The calculation of percentage of moisture in the butter may be made according to example given in connection with the gravimetrical method.

Other scales, sensitive to one milligram, may be substituted for the chemical balance where conditions are observed as described above. By using exactly ten grams, the percentage water content may be calculated more easily.

Comparisons of Results Obtained by the Official and Richmond Methods.

No.	Official Method	Richmond Method	Difference	No.	Official Method	Richmond Method	Difference
1	15.90	15.85	—.05	2	12.94	13.22	.28
3	15.45	15.86	.41	4	15.27	15.39	.12
5	14.16	14.35	.19	6	14.90	14.96	.06
7	19.10	19.18	.08	8	16.38	16.52	.14
9	18.27	18.33	.06	10	15.95	15.97	.02
11	17.32	17.58	.26	12	19.25	19.46	.21
13	14.81	14.98	.17	14	17.68	17.74	.06
15	16.96	16.86	—.10	16	15.50	15.55	.05
17	14.68	15.14	.46	18	13.62	13.80	.18
19	13.38	13.27	—.11	20	14.86	14.99	.13
21	13.80	13.92	.12	22	12.91	13.14	.23
23	14.02	14.14	.12	24	13.17	13.24	.07
25	12.94	13.02	.08	26	13.63	13.65	.02
27	11.07	11.12	.05	28	14.20	14.28	.08
29	14.66	14.32	—.34	30	15.17	15.27	.10
31	14.26	14.64	.38	32	12.06	12.14	.08
33	13.88	13.45	—.43	34	14.10	14.06	—.04
35	13.64	13.74	.10	36	14.82	14.80	.02
37	14.08	13.97	—.11	38	13.71	13.67	—.04
39	12.41	12.69	.28	40	13.30	13.34	.04
41	11.77	11.76	—.01	42	14.60	14.64	.04
43	14.88	15.09	.21	44	12.62	12.71	.09
45	15.24	15.02	—.22	46	14.27	14.45	.18
47	16.53	16.54	.01	48	16.42	16.67	.25
49	16.03	16.20	.17	50	16.42	16.39	—.03
51	13.57	13.58	.01	52	14.34	14.35	.01
53	13.44	13.41	—.03	54	15.98	15.88	—.10
55	14.16	14.33	.17	56	14.30	14.46	.16
57	14.54	14.73	.19	58	14.80	14.70	—.10
59	14.53	14.66	.13	60	14.60	14.61	.01
61	15.	15.35	.35	62	14.72	14.66	—.06
63	15.59	15.81	.22	64	12.84	12.86	.02
65	12.66	13.32	.56	66	12.99	13.18	.19
67	15.85	15.87	.02	68	15.54	15.67	.13
69	15.97	16.15	.18	70	15.46	15.44	.02
71	16.51	16.40	—.11	72	15.86	15.89	.03
73	16.52	16.63	.11	74	14.79	14.84	.05
75	14.86	14.86	.00	76	12.55	12.76	.21
77	13.20	13.47	.27	78	16.84	16.80	—.04
79	16.32	16.25	—.07	80	15.79	15.90	.11
81	16.09	16.42	.33	82	16.60	16.75	.15
83	17.60	17.49	—.11	84	15.22	15.38	.16
85	14.16	14.28	.12	86	15.26	15.18	—.08
87	15.64	15.72	.08	88	15.68	15.88	.20
89	14.71	14.78	.07	90	15.06	15.24	.18
91	15.68	15.75	.07	92	15.71	15.54	—.17
93	15.75	15.80	.05	94	13.72	14.04	.32
95	14.00	13.84	—.16	96	13.62	13.77	.15
97	15.47	15.63	.16	98	16.36	16.45	.09
99	15.06	15.18	.12	100	16.65	16.62	—.03

The above results show that out of 100 samples analyzed 21 samples show a difference of over .2%, nine of these show a

difference of over .3%, and three exceed .4% in error. Three samples analyzed by the Richmond Method show a water content more than .2% less than that obtained by the Official Method. Many show a difference of less than one tenth of one per cent and in quite a number of cases the difference may be reckoned in the hundredths of one per cent.

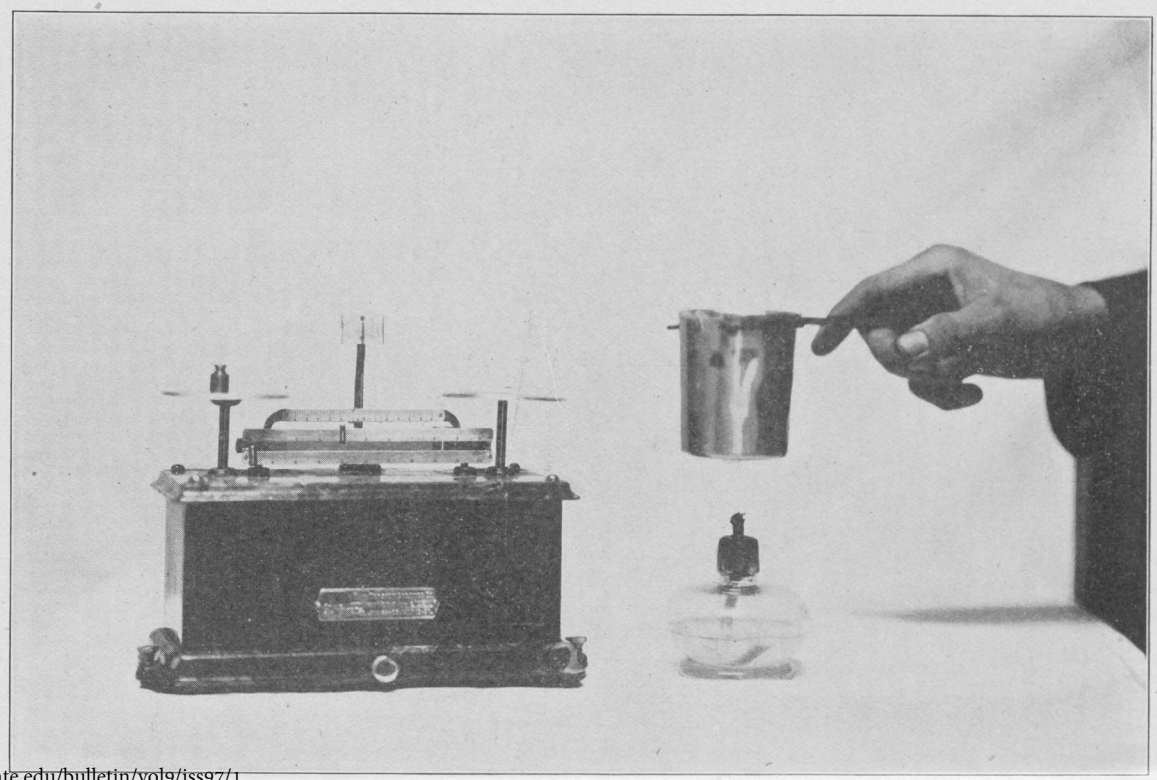
As a safeguard in keeping the water content below the standard fixed by law, the Richmond Method is to be recommended. There is very small chance of the analysis showing a lower percentage moisture content than it actually contains. In other words if the analysis of sample shows 16% water content it may be depended upon that there is not any more than 16% moisture though there is a chance of it being slightly less.

ALUMINUM BEAKER METHOD.

This method was devised by George E. Patrick, Chief Dairy Laboratory of the Department of Agriculture, Washington, D. C. In general it may be said to be a modification of the Richmond Method. The use of the aluminum beaker, in place of the porcelain or aluminum dishes commonly used, and shaking with a rotary motion in place of stirring with a rod, are the main features that distinguish it from the method advocated by Richmond.

In brief, the method consists of weighing a sample of butter into an aluminum beaker and heating it over the flame of an alcohol lamp. A cone shaped asbestos chimney, about 6 inches high and well ventilated at the base, is used to concentrate the heat and prevent deposition of soot upon the bottom of the beaker. The beaker is kept in constant rotation to prevent overheating the butter. The sides of the beaker are not allowed to reach a temperature at which 'sizzling' is produced when they are touched with the moistened finger. After the greater part of the water is expelled and foaming has ceased the sides of the beaker are heated to sizzling temperature and the foam thrown upon them by a lively rotation. The bottom of the beaker is again reheated gently until all the water is expelled.

Either a chemical balance or other scales may be used. Metric weights from ten grams to one centigram are required. The water is driven off as described above, the beaker is then cooled by sinking it nearly to the rim in water, wiped dry, replaced immediately upon the balance, and brought again to equipoise by adding weights to the same side to replace the weight of the water lost. The weight required in grams, multiplied by 10 equals the percentage water. If it requires two grams to replace in weight the water evaporated, the moisture content would be 20%; if one gram, four decigrams, two centigrams or 1.42 grams were required the moisture content would be 14.2%. Similarly if one gram, five decigrams, nine centi-



USING THE ALUMINUM BEAKER.

grams, or 1.59 grams were required the percentage water content would be 15.9.

For renovated butters it is recommended to use a glass stirring rod to prevent the gathering of the caseous matter into pellets. More careful heating is necessary to prevent mechanical losses through violent ebullition of sample.

The results as given by Patrick are very favorable to this method. Out of 42 samples, in only two cases does the difference between the Official and Aluminum Beaker Method exceed the "limit of error" allowed in the chemical analysis of butter. The use of the aluminum beaker has the advantage of construction in that there would be little or no danger of sputtering of fat. The higher sides of beaker would prevent this. Being aluminum it would not readily be broken. In this it would have an advantage over porcelain and other breakable dishes. The use of 10 grams as the weight of sample makes it more simple in calculation of percentage. Below are given the results obtained at this Station.

Comparisons of Results Obtained by the Official and Patrick Tests.

No.	Official Method	Patrick Method	Difference	No.	Official Method	Patrick Method	Difference
1	15.90	16.25	.35	2	12.94	14.5	1.56
3	15.45	16.2	.75	4	15.27	15.6	.33
5	14.16	15.0	.84	6	14.90	15.0	.10
7	19.10	19.8	.70	8	16.38	16.8	.42
9	18.27	19.1	.83	10	15.95	16.2	.25
11	17.32	18.	.68	12	19.25	19.5	.25
13	14.81	15.5	.69	14	17.68	18.1	.42
15	16.96	17.5	.54	16	15.50	16.1	.60
17	14.68	15.2	.52	18	13.62	13.8	.18
19	13.38	13.2	—.18	20	14.86	15.0	.14
21	13.80	14.0	.20	22	12.91	13.5	.59
23	14.02	14.0	—.02	24	13.17	13.8	.63
25	12.94	13.	.06	26	13.63	14.5	.87
27	11.07	11.5	.43	28	14.20	15.6	1.40
29	14.66	14.5	—.16	30	15.17	15.	—.17
31	14.26	15.	.74	32	12.06	12.2	.14
33	13.45	14.0	.55	34	14.10	14.2	.10
35	13.64	13.9	.26	36	14.82	15.3	.48
37	14.08	14.0	—.08	38	13.71	13.8	.09
39	12.41	12.3	—.11	40	13.30	13.0	—.30
41	11.77	12.3	.53	42	14.60	15.2	.60
43	14.88	16.0	1.12	44	12.62	13.0	.38
45	15.24	15.0	—.24	46	14.27	14.5	.23
47	16.53	16.0	—.53	48	16.42	16.	—.42
49	16.03	16.0	—.03	50	16.42	16.	—.42
51	13.57	14.0	.43	52	14.34	14.0	—.34
53	13.44	13.0	—.44	54	15.98	16.0	.02
55	14.16	14.0	—.16	56	14.30	14.8	.50
57	14.54	14.0	—.54	58	14.70	15.0	.30
59	14.53	14.3	—.23	60	15.59	16.2	.61

These results were obtained by stirring with glass rod similar to Richmond Method.

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No.	Official Method	Patrick Method	Difference	No.	Official Method	Patrick Method	Difference
1	15.85	16.0	.15	2	15.54	15.7	.16
3	15.97	16.5	.53	4	15.46	15.3	—.16
5	16.51	16.2	—.31	6	15.86	16.1	.24
7	16.52	16.4	—.12	8	14.79	15.3	.51
9	14.86	15.0	.14	10	12.55	12.5	—.05
11	13.20	12.5	.30	12	16.84	17.5	.66
13	16.32	16.2	—.12	14	15.79	16.0	.21

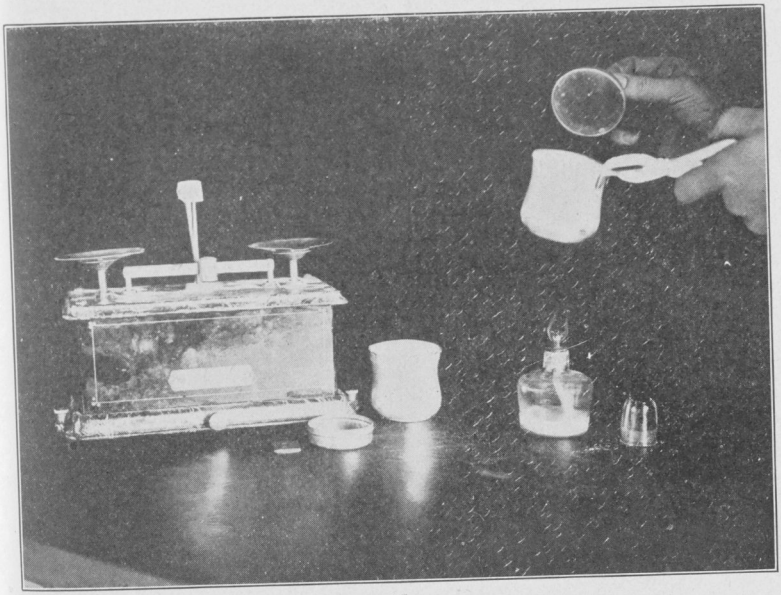
In the first series of results the analysis shows a very marked irregularity. An excess of one per cent is not uncommon while often this excess approaches 1.5 per cent. Often the results compare exactly with those obtained by the Official Method while others are within .2%. In some instances the results are low, in other instances they are much in excess of those obtained by the Official or Gravity Method. So much so, that no dependence may be placed upon them. It was thought that better results might be obtained where a glass rod was used as a stirrer as in the Richmond Method. The results, as given above show up more favorably though out of the fourteen analyses four exceed the results obtained by the Official Method by .5%.

It would seem then that with the use of the aluminum beaker, the heat is too readily conducted to the butter. When placed over a flame certain portions of the sample are thus heated sufficiently high to allow burning of the fat. The percentage of water so obtained is too high. When heated more slowly by placing beaker on wire gauze or on asbestos board there is the difficulty of telling by cessation of foaming when all the moisture is driven off. In the use of the Richmond Method with a little practice one may readily determine this by this means. The foaming accompanied by a crackling sound ceases suddenly and is easily recognized. Where heat is applied more slowly it is much more difficult, if not impossible, to determine when to stop heating. The porcelain dish distributes the heat to the sample more evenly than the aluminum vessel. In the use of the Richmond Method, also, there was much less certainty of accurate results where an aluminum dish was used.

THE IRISH METHOD.

In principle the Irish Method is the same as Patrick's Method. The distinctive characters are: first, the use of a mirror to show when steaming has ceased; second, a set of weights for the dried sample that show percentage of water directly. The mirror is not to be wholly relied upon to show when all the water is driven off. In a dry warm atmosphere the

moisture may be driven off the sample and be immediately absorbed. On the other hand when the air is saturated with moisture, as it is in some creameries, it is a difficult matter to



IRISH METHOD.

determine just when moisture ceases to be expelled from the sample by the heating process.

Comparisons of Results Obtained by the Official and Irish Tests:

No.	Official Method	Irish Method	Difference	No.	Official Method	Irish Method	Difference
1	15.90	16.35	.45	2	12.94	14.3	1.36
3	15.45	15.75	.30	4	15.27	15.6	.33
5	14.16	14.4	.24	6	14.90	14.9	.00
7	19.10	19.45	.35	8	16.38	16.8	.42
9	18.27	19.1	.83	10	15.95	16.1	.15
11	17.32	17.8	.48	12	19.25	19.8	.55
13	14.81	15.4	.59	14	17.68	17.9	.22
15	16.96	17.5	.54	16	15.50	16.0	.50
17	14.68	15.1	.42	18	13.62	13.5	—.12
19	13.38	13.5	.12	20	14.86	15.0	.14
21	13.80	14.0	.20	22	12.91	13.4	.49
23	14.02	14.	—.02	24	13.17	13.5	.33
25	12.94	13.	.06	26	13.63	14.3	.67
27	11.07	11.25	.18	28	14.20	15.5	1.30
29	14.66	14.5	—.16	30	15.17	15.4	.23
31	14.26	15.	.74	32	12.06	12.0	—.06
33	13.88	14.0	.12	34	14.10	13.8	—.30
35	13.64	13.6	—.04	36	14.82	15.1	.28
37	14.08	14.0	—.08	38	13.71	13.8	.09
39	12.41	12.5	.09	40	13.30	13.0	—.30
41	11.77	12.5	.73	42	14.60	15.0	.40
43	14.88	16.2	1.32	44	12.62	13.0	.38
45	15.24	15.5	.26	46	14.27	14.9	.63
47	16.53	17.5	.97	48	16.42	16.	—.42
49	16.03	16.8	.77	50	16.42	16.	—.42
51	13.57	14.0	.43	52	14.34	14.	.34
53	13.44	13.2	—.24	54	15.98	15.5	—.48
55	14.16	14.0	—.16	56	14.30	14.5	.20
57	14.54	14.0	—.54	58	14.70	14.7	.00

In the above table very many results are alike. Along with these however are found three which exceed the gravity by one per cent and a much larger number which show a difference of over .5%.

For reasons mentioned in connection with the Patrick Method, the aluminum vessel may not be used with any degree of accuracy.

Comparison of Results Obtained by the Official Method and the Irish Method.

No.	Official Method	Irish Method	Difference	No.	Official Method	Irish Method	Difference
1	16.40	16.4	.00	2	16.05	15.8	—.25
3	16.08	16.0	—.08	4	14.55	14.6	.05
5	12.77	12.9	.13	6	12.88	13.10	.22
7	13.85	13.8	—.05	8	14.65	14.6	—.05
9	13.11	12.9	—.21	10	14.69	15.0	.31
11	15.29	15.2	—.09	12	12.89	13.1	.21
13	13.34	13.6	.26	14	12.42	12.8	.38
15	14.38	14.4	.02	16	13.65	14.0	.35
17	13.81	14.5	.69	18	13.12	12.7	—.42
19	13.80	14.0	.20	20	13.30	13.6	.30
21	12.87	13.4	.53	22	13.45	13.7	.25
23	14.30	14.3	.00	24	13.76	13.8	.04
25	13.51	14.0	.49	26	13.44	13.9	.46
27	13.48	13.7	.22	28	11.84	11.8	.04
29	14.57	14.7	.13	30	12.28	12.4	.12
31	13.41	13.4	—.01	32	13.23	13.3	.07
33	13.12	13.3	.18	34	11.65	12.2	.55
35	11.22	11.3	.08	36	13.17	13.7	.53
37	12.58	12.8	.22	38	12.62	12.6	—.02
39	14.88	15.2	.32	40	14.88	15.1	.22
41	12.74	13.11	.37	42	12.75	13.1	.35
43	14.97	15.1	.13	44	14.25	14.0	—.25
45	15.84	16.3	.46	46	13.52	13.9	.38
47	14.95	14.9	—.05	48	14.02	14.4	.38
49	15.53	15.8	.27	50	14.15	14.9	.75
51	14.89	15.5	.61	52	12.83	13.6	.77
53	12.28	13.0	.72	54	14.27	14.2	—.07
55	11.74	12.8	1.06	56	11.48	12.7	1.12
57	14.69	15.6	.91	58	11.39	12.2	.81
59	14.92	16.2	1.28	60	11.75	12.6	.85
61	14.30	15.5	1.2	62	12.26	13.3	1.04

When a sample is heated directly over the flame, burning is certain to occur, particularly where the heating period is extended beyond the time required to expel the moisture. The following trial was made. The samples were first heated till all moisture was driven off, and percentage moisture content calculated. The same sample was then heated for another minute and loss of weight presented as percentage moisture content. The sample was again submitted to a third heating of a minute's duration and loss of weight recorded as in the first and second case. The results are presented in the following table.

Table Showing Effects of Extended Heating:

No.	Per Cent Moisture	Per ct. moisture 2nd trial	Increase	Per ct. moisture 3rd Trial	Further Increase	Total Increase
1	10.	10.4	.4	10.7	.3	.7
2	13.5	13.8	.3	14.3	.5	.8
3	18.0	18.5	.5	19.0	.5	1.0
4	9.1	9.4	.3	9.6	.2	.5
5	11.6	12.2	.6	12.6	.4	1.0
6	14.5	14.8	.3	15.2	.4	.7
7	16.6	17.0	.4	17.4	.4	.8
8	11.8	12.5	.7	12.9	.4	1.1
9	13.5	14.0	.5	14.5	.5	1.0
10	14.5	15.2	.7	15.8	.4	1.1

The above table goes to show the reason of some of the irregularities of those methods where heat is applied directly to dish containing sample.

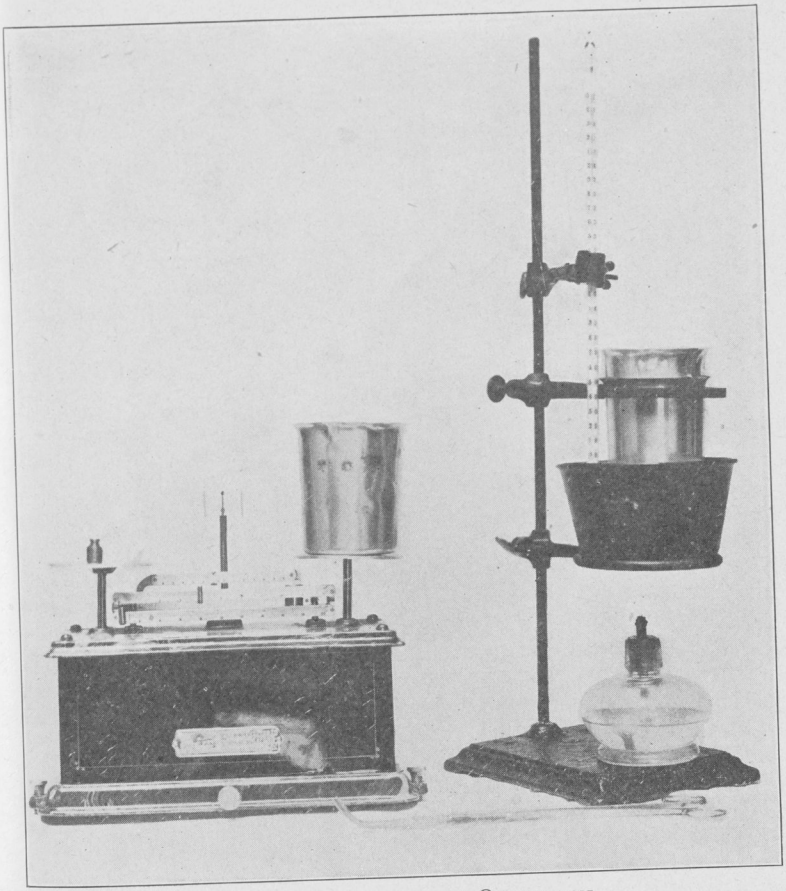
THE AMES METHOD.

To overcome inaccuracies involved in the use of the aluminum beaker, due to lack of control of heating temperature, the Dairy Section of this Experiment Station conceived the idea of using as a controlling factor a liquid with a boiling point considerably higher than water. This requirement was fulfilled by using paraffin. It was first used by Mr. Brown.

A vessel containing paraffin is heated over a flame to a temperature ranging between 150 and 200 degrees. Best results were obtained where a temperature approaching 175 degrees was employed. Ten grams of butter are weighed into a suitable vessel,—the aluminum beaker used in the Patrick and Irish methods may be used,—and placed in the heated paraffin until all foaming ceases. During the heating process the butter should be occasionally shaken. Care should be taken to have the paraffin at desired temperature before placing in it the vessel containing the sample. After heating, the outside of vessel it should be wiped carefully with a dry cloth to remove any paraffin that may adhere. The beaker and sample after being cooled is reweighed and percentage water content may then be calculated. The heating process requires about five minutes. Either a chemical balance or other suitable scale may be used.

To overcome the objection that may be raised to heating the vessel in paraffin direct, two beakers may be used, one fitting closely inside the other. Either aluminum or copper beakers may be used. The beaker or vessel containing the sample could then be placed in the outer or larger one and the latter would come in contact with the paraffin. This would overcome the necessity of wiping the vessel containing the sample, and would avoid any error from this source. If any

paraffine is allowed to remain on vessel or if care is not taken to use a dry clean cloth the results are liable to be low.



THE AMES METHOD IN OPERATION.

The following results were obtained using this method:

THE AMES METHOD.

No.	Official Method	Ames Method	Difference	No.	Official Method	Ames Method	Difference
1	15.61	15.8	.19	2	10.84	10.8	—.04
3	10.55	10.6	.05	4	13.95	14.0	.05
5	8.47	8.75	.28	6	12.55	12.7	.15
7	13.33	13.4	.07	8	15.09	15.0	—.09
9	15.26	15.4	.14	10	15.82	15.8	—.02
11	15.23	15.2	—.03	12	14.95	15.1	.15
13	14.08	14.1	.02	14	13.25	13.3	.05
15	14.74	14.8	.06	16	14.20	14.3	.10
17	12.04	12.15	.11	18	12.48	12.6	.12
19	10.92	10.8	—.12	20	12.04	12.1	.06
21	11.01	11.0	—.01	22	13.99	14.3	.31
23	14.72	15.	.28	24	13.12	13.1	—.02
25	14.71	15.	.29	26	17.38	17.5	.12
27	15.21	15.20	—.01	28	14.04	14.00	—.04
29	15.90	16.05	.15	30	15.34	15.2	—.14
31	13.10	13.4	.30	32	14.22	14.3	.08
33	12.30	12.35	.05	34	16.09	16.3	.21
35	15.19	15.2	.01	36	17.16	17.1	—.06
37	15.08	15.1	.02	38	14.30	14.3	.00
39	19.35	19.5	.15	40	16.82	16.8	—.02
41	16.52	16.5	—.02	42	16.90	17.	.10
43	16.40	16.5	.10	44	11.94	12.0	.06
45	14.06	14.0	—.06	46	16.60	16.55	—.05
47	14.71	14.7	—.01	48	18.00	18.0	.00
49	14.71	14.7	—.01	50	13.72	13.7	—.02

The above table shows six samples which vary .2% from the Official Method; of these six, one only exceeds .3% difference. As mentioned, the aluminum beaker used in the Patrick and Irish Methods may readily be used. It would require the purchase of some suitable vessel to heat the paraffin and also a thermometer to determine the higher temperature. As stated above there is the possibility of burning of fat where sample is exposed too long to the high temperature. Where temperature control is obtained this is not so apt to occur. We submit the following table.

GRAPHIC COMPARISON OF RESULTS.

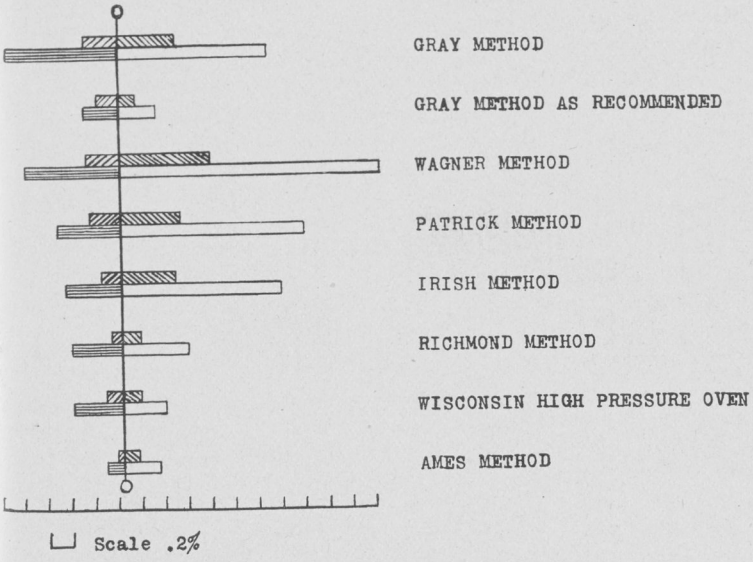
AVERAGE DIFFERENCE OF RESULTS ABOVE OFFICIAL METHOD

AVERAGE DIFFERENCE OF RESULTS BELOW OFFICIAL METHOD

MAXIMUM DIFFERENCE ABOVE OFFICIAL METHOD

MAXIMUM DIFFERENCE BELOW OFFICIAL METHOD

O–O OFFICIAL METHOD



No.	Temperature	% Moisture 1st heating till foaming ceased	% Moisture 2d heating of five min.	% Moisture 3d heating of five min.
1	170 C	15.0	15.0	15.0
2	160	14.5	14.5	14.5
3	160	14.3	14.3	14.3
4	160	12.0	12.0	12.0
5	165	12.0	12.0	12.0
6	160	13.6	12.0	12.0
7	175	13.8	14.0	14.0
8	150	21.0	21.0	21.0
9	175	13.8	13.8	13.8

The above table shows that there is little danger of heating sample too long provided temperature is controlled. Sam-

ple No. 7 shows an increase of .2%. This may have been because first heating was not continued quite long enough to evaporate all moisture in sample. The third heating did not show any further increase. It is evident that possible burning of fat is an almost negligible factor with this method. This should be true also in the use of the Wisconsin High Pressure Oven.

Below is found a further table of comparisons of the "Ames" and "Official" Methods. This table represents the work of J. Bower. Samples were heated to a temperature of from 170 to 185 degrees C. for exactly five minutes. Further heating failed to show an increased loss of weight. A Torsion balance No. 1700 was used. In the use of this balance it was found necessary to place vessel containing sample the same place on sample pan at each weighing. Variations of .1% or even greater may be obtained by failing to observe this point.

No.	Official Method	Ames Method	Difference	No.	Official Method	Ames Method	Difference
1	12.14	12.00		2	20.21	20.35	
	12.09	12.00	.11		20.23	20.40	.15
3	13.62	13.50		4	9.30	9.30	
	13.62	13.50	.12		9.32	9.30	.01
5	14.17	13.95		6	14.47	14.40	
	14.28	14.00	.25		14.51	14.50	.04
7	12.20	12.20		8	25.37	25.50	
	12.23	12.30	.04		25.29	25.50	.17
9	22.84	23.10		10	15.18	15.30	
	22.91	23.00	.18		15.30	15.40	.11

A further experiment was conducted using the chemical balance. The following table shows the results obtained:

No.	Official Method	Ames Mthd 1st Heating 5 minutes	Difference	Ames Mthd 2nd Heating 5 minutes	Difference	Ames Mthd 3rd Heating 5 minutes	Difference
1	17.92	17.92	.00	17.92	.00	17.95	.03
	17.74	17.80	.06	17.90	.16	17.86	.12
2	14.43	14.56	.13	14.62	.19	14.58	.15
	14.43	14.65	.22	14.72	.29	14.66	.23
3	14.31	14.41	.10	14.48	.17	14.50	.19
	14.54	14.43	—.11	14.49	—.05	14.44	—.10
4	16.09	16.23	.14	16.32	.23	16.28	.19
	15.99	16.16	.17	16.15	.16	16.12	.13
5	16.57	16.54	—.03	16.63	.06	16.63	.06
	16.60	16.62	.02	16.68	.08	16.63	.03
6	9.74	9.86	.12	9.90	.16	9.94	.20
	9.71	9.71	.00	9.78	.07	9.80	.09
7	12.75	12.85	.10	12.94	.19	12.95	.20
	12.73	12.89	.16	12.98	.25	12.99	.26
8	14.11	14.28	.17	14.31	.20	14.32	.21
	14.10	14.38	.28	14.36	.26	14.38	.28
9	13.00	13.10	.10	13.22	.22	13.25	.25
	13.15	13.25	.10	13.25	.10	13.28	.13
10	14.15	14.11	—.04	14.23	.08	14.24	.09
	14.13	14.25	.12	14.25	.12	14.28	.15
11	11.35	11.43	.08	11.42	.07	11.42	.07
	11.24	11.27	.03	11.39	.15	11.40	.16
12	12.08	12.07	—.01	12.21	.13	12.24	.16
	12.10	12.26	.16	12.31	.21	12.27	.17
13	11.40	11.53	.13	11.55	.15	11.59	.19
	11.44	11.45	.01	11.46	.02	11.49	.05
14	12.42	12.53	.11	12.56	.14	12.56	.14
	12.40	12.46	.06	12.55	.15	12.56	.16
15	14.03	14.05	.02	14.17	.14	14.11	.08
	14.05	14.10	.05	14.17	.12	14.16	.11
16	15.15	15.20	.05	15.30	.15	15.23	.08
	15.16	15.23	.07	15.30	.14	15.29	.13
17	14.34	14.54	.20	14.64	.30	14.54	.20
	14.34	14.56	.22	14.55	.21	14.58	.24
18	11.67	11.80	.13	11.82	.15	11.88	.21
	11.71	11.78	.07	11.90	.19	11.90	.19
19	17.78	17.70	—.08	17.79	.01	17.82	.04
	17.73	17.74	.01	17.75	.02	17.85	.12
20	15.49	15.52	.03	15.54	.05	15.59	.10
	15.54	15.56	.02	15.58	.04	15.59	.05

REVIEW OF OTHER METHODS.

CAROLL'S TESTER

This apparatus consists of a special measure for butter: color glass tubes in which the butter is melted and the separated water measured. The tubes are placed in boiling water for about forty-five minutes being occasionally removed and shaken. By this means it is claimed the water will be separated and collected in the lower part of the tubes. After many

trials this method proved to be valueless and results not even approximately correct, variations of over 3% being not uncommon. This is similar to results obtained at Ottawa, Canada.*

We quote the following:

	Moisture	
	By Gravimetrical Analysis	By Carell's Tester
Sample A, print butter, C. E. F.	13.76	9.0
		8.0
		5.0
Sample B, print butter, C. E. F.	13.13	6.0
		4.0

THE GELDARD BUTTER TESTER.

This method is similar to that recommended by Richmond.** It differs only in amount used. Fifty grams of butter are weighed into a small porcelain dish together with a metal stirrer and a small quantity of attenuating material. This is submitted to a temperature that will result in evaporation of water without burning of the fat. A report on this method is given above under the Richmond Method. *The Ottawa Station presents the following data:

	Moisture	
	By Analysis	By Geldard's Apparatus
		13.6
Sample A, print butter, C. E. F.	13.76	13.8
		13.6
		13.2
Sample B, print butter, C. E. F.		13.2
	13.13	13.2

"These results are extremely satisfactory, and show that the method is capable of furnishing data in close accordance with those obtained by accepted methods of analysis."

THE WAGNER BUTTER HYGROMETER.

This piece of apparatus, before the introduction of the Gray and other tests, was used considerably in creamery practice. Of late it has gradually given place to these latter tests. While in use it gave very uncertain results. At this Station it was found to be of no value in the determination of water in butter.

According to printed circular accompanying the bottles, 18 grams of butter are weighed into graduated test tube, the

*Bulletin No. 6, Dept. of Agr., Ottawa, Can.
**Page 251, Dairy Chemistry, Henry Droop Richmond.

tube being closed by the soft rubber stopper and then inserted in the water bath cylinder at about 140 degrees F., the graduated test tube being held in position by a soft rubber support. As soon as the butter has melted completely the apparatus is placed in a Babcock testing machine and whirled for about 10 minutes. If hand Babcock testing machine is used, the water bath should be reheated two or more times during whirling. The water content of the butter will soon collect at the bottom of the graduated test tube and can be read directly from the scale. In the case of salted butter 2% should be allowed for salt. It will be observed that there is a sharp layer of water as well as a sharp layer of casein (the casein is combined with water). Every 1% combined water and casein indicated on the hygrometer should be read 0.1 per cent casein. We have come to this result by removing the casein of the combined casein and water by drying same. For instance, if the butter hygrometer shows:

A sharp water line of.....6%
A sharp combined casein and water line of.....11%
The moisture would be.....15.9%
Casein if dried to powder.....1.1
Actual butter-fat.....83.

It is difficult to determine the line of demarcation with such an apparatus. From the graduation it is difficult to read closer than 1%. This results in giving only approximate results. The percentage of salt may readily vary from 1 to 5%.

Professor Shutt of the Experiment Station, Ottawa, Canada, writes the following:

"The writer, after considerable experience with this hygrometer, cannot speak in unqualified terms as to its general satisfactoriness. It is quite true that in a number of trials the readings, after calculations, gave data sufficiently near the true water content for all practical purposes, but the uncertainty in obtaining distinct layers which can be readily read off seems to be too great to make the instrument of value in the warehouse or dairy, where it is particularly desirable that the readings should not only be fairly accurate, but also easily and quickly made."

Similar to the above method is the method used by some makers of making a fat determination by means of cream test bottle or special butter bottle. To the reading thus obtained is added a percentage which is supposed to approximate the percentage composition of caseous matter, salt and ash. The total is subtracted from 100 and the remainder is taken as representing the percentage of water in butter.

Example.
Butter-fat 82.5
Caseous matter, salt and ash..... 3.0

Total 85.5
Percent Water Content 14.5

Such a method is even more erroneous than the one previously described. The determination of fat by means of cream test bottles or special bottles, unless very carefully conducted, is not to be relied upon as a correct method of determining the fat content of butter. Incorrect calibration, expansion and contraction of glassware through temperature changes, temperature effects on the volume of fat in bottle make it highly improbable that results will compare favorably with chemical analysis. When the variation of both salt and caseous matter as it is found in butter, is further considered, the utter unreliability of such a method is apparent.

SAMPLING OF BUTTER.

In the analysis of butter for water the importance of obtaining a correct sample can not be too strongly emphasized. Many of the results now obtained by makers and others are unreliable because the sample does not contain the constituents of the butter in the same proportion as the butter that is being analyzed. If it is a sample of a churning that is to be analyzed it has been our practice to take a number of samples, from 10 to 20 grams each, from different portions of the churning as the butter is removed from the churn. These small samples may be taken by a spatula and placed in a Mason or other suitable jar. Samples may also be taken by a butter trier or sampler, care being taken to get samples which, as a composite, are representative of the whole churning and not of one portion only. The necessity of this will be explained below.

In sampling from a tub, the butter should be held in a refrigerator until firm enough to be readily sampled by a butter trier. In using a trier it is best to take the sample in a diagonal direction the full depth of the tub. The sample may then be transferred carefully to sample vessels, care being taken that none of the water is lost in the transfer. If a sample is taken from two other tubs in the same manner a composite sample representative of a large churning may thus be obtained. Samples of prints or other packages of butter can best be obtained by the use of the butter trier.

SELECTION AND CARE OF SCALES.

Too many of the scales at present used for weighing samples are not sensitive enough to give anything like satisfactory results. The ordinary cream scales are in many cases not to be depended upon for this work. A special scale sensitive to at least one milligram should be used, while a chemical balance should be sensitive to at least one half milligram. Considerable care should be given them. Too often the cream scales, through exposure to dampness of creamery or careless handling, are utterly unfit for use. In using any uncovered scale

great care should be exercised to avoid draughts as a variation of from .2 to 1% may be easily attributed to this cause alone.

Analysis of butter is usually desired on completion of churning. Samples for analysis are either taken from churn or from tubs the following day. Under present competition there would be little need to make a moisture determination before completion of churning. Should the maker fear that he has exceeded the limit of moisture content allowed by law, a rapid method of determining the amount of water would be of practical benefit to him. Should the churning show an excess of mosture, he could by certain methods known to practical makers reduce the water content somewhat.

PREPARATION OF SAMPLE.

In preparing the samples for analysis it is essential that any portion taken shall be representative. Under "Methods for the Analysis of Dairy Products"* the preparation of sample is given as follows:

"If large quantities of butter are to be sampled, a butter trier or sampler may be used. The portions thus drawn, about 500 grams, are to be perfectly melted in a closed vessel at as low a temperature as possible, and when melted the whole is to be shaken violently for some minutes until the mass is homegeneous, and sufficiently solidified to prevent the separation of the water and fat."

Another method used by dairy chemists is to take a sample of butter and place it in a suitable container (1 pt. Mason jar will be satisfactory,) This container is placed in water at about 100 degrees F. The butter is stirred with a spatula or spoon until it is about the consistency of thick cream and no free water can be seen. Samples of butter should not be left standing in open containers any length of time before making water determination, as some of the moisture will evaporate and the percentage of water shown when the determination is finally made will be too low.

This second method has been found to give satisfactory results provided the butter is stirred sufficiently to get an even distribution of the several constituents. Whatever method is employed this distribution should be thorough. Many irregularities in results obtained through careless preparation of sample can be avoided in this way.

Even where care is taken it is found that in duplicate tests from the same samples there may be variation in results. Butter, being a mechanical mixture of which water and fat are constituent parts, presents certain difficulties not found in preparation of samples of other substances. Where butter is melted, the tendency is for the different constituents to separate according to difference in specific gravity. It requires

*Bulletin 46, pp 43, U. S. Dept. of Agr.

vigorous shaking during the cooling process to obtain a homogeneous mass. When prepared at the lower temperature, through mixing must be made. In no case, however, need the variation exceed .2 of 1%, if directions be followed carefully. In fact such a variation is an exception in the hands of a careful worker, though occasionally a slightly greater variation is obtained.

Differences in moisture content greater than the above may be due to causes other than those explained above. Often times samples from same churn or from same tub vary in per cent water content. Experiments to better explain these differences are here given.

WATER IN BUTTER FROM DIFFERENT PARTS OF THE CHURN.

No.	Drain End	Middle	Gear End	Average
1	15.10	14.20	14.66	14.67
2	14.10	13.64	13.88	13.87
3	14.30	14.16	14.54	14.33
4	15.59	14.72	15.00	15.10
5	12.67	12.66	12.99	12.74
6	15.54	15.97	15.46	15.65
7	16.51	15.86	16.52	16.29
8	16.84	16.32	15.79	16.31
9	16.09	16.60	17.60	16.76
10	15.22	14.16	15.26	14.88
11	15.68	15.71	15.75	15.71
12	13.72	14.00	13.62	13.78
13	16.65	16.45	16.18	16.42
14	13.45	12.42	13.49	13.12
15	17.54	15.83	15.22	16.19
16	15.20	15.41	15.48	15.36
17	14.19	14.16	14.17	14.17
18	15.07	14.87	14.60	14.84
19	15.72	16.01	15.68	15.80
20	13.62	13.85	13.68	13.85
21	14.70	14.42	14.43	14.51
22	15.93	15.04	15.01	15.32
23	17.06	15.86	16.22	16.38
24	14.95	14.30	16.08	15.11
25	16.84	16.32	15.79	16.31
26	16.09	16.60	14.71	15.80
27	15.22	15.38	15.41	15.33
28	15.68	15.71	15.75	15.71
29	16.65	16.45	16.18	16.42
30	13.45	12.42	13.49	13.12
31	17.52	15.83	15.22	16.10
32	15.20	15.41	15.48	15.36
33	15.07	14.87	14.60	14.84
34	14.70	14.42	14.43	14.51
35	15.93	15.04	15.01	15.32
36	17.06	15.86	16.22	16.38

The above results were obtained from samples taken from the Victor churn on the completion of churning. In this style

of churn, unless maker moves part of the butter from ends of churn toward the center, the tendency is for the butter to accumulate towards the end of churn. As a result it is found that the butter from ends of churn shows a higher moisture content than that from center. In some of the above numbers there is a uniformity of results that is lacking in others. A sample from one portion of the churning is not then representative of that churning. It is essential, as was mentioned above, to take a number of samples from different parts of the churning if an accurate sample is to be obtained. The average in the above table would more nearly represent the water content in the churnings.,

If this is true of a churning, it is also the case of tubs from a churning. This would be particularly so where, as is sometimes practiced, tubs are filled one by one, and where each tub would be taken from one part of the churning. Any one tub would not be representative of the churning and the analysis of a sample from a single tub could not be taken to represent the percentage moisture content of that particular churning. Should the butter, during the working process, be kept uniformly proportioned above the rolls and butter be packed into several tubs in such a manner as to distribute smaller portions of the churning in succession to each tub, the sample would then be more uniform. Smaller churnings and other types of churns may give different results from those given above. An attempt was made by the churner to obtain a more even distribution of moisture throughout the body of the butter in the churn as described above. The following table is here given:

Results from Victor churn with composite sample of churning and from tub following day.

No.	Drain End	Middle	Gear End	Average	Composite	Tub
1	12.71	12.46	12.41	12.52	12.58	11.78
2	13.94	13.50	13.48	13.64	13.56	13.53
3	12.96	13.77	13.21	13.31	13.54	12.57
4	12.52	11.89	12.01	12.14	12.20	12.17
5	12.77	12.87	12.34	12.66	12.66	12.25
6	13.16	13.10	13.05	13.10	12.98	12.68
7	13.79	14.05	14.00	13.94	14.15	13.93
8	12.29	11.64	11.06	11.66	11.55	11.36
9	13.21	13.89	13.26	13.45	13.63	13.25
10	12.43	12.95	12.75	12.71	1284.	11.64
11	13.30	13.33	13.74	13.45	13.36	12.98
12	14.93	14.16	14.66	14.56	14.03	14.28
13	14.77	14.31	14.70	14.59	14.85	14.13

In this table is noticed a little more regularity in the moisture content in samples from different parts of the churn. The moisture content of composite sample compares closely

with that obtained by a calculation of the average of the other three samples. The sample taken from the tub, while in some cases practically the same as that of an average or composite sample, in other instances vary from .1% to over 1%. This may be due to two causes; first, sampling; second, to expulsion of moisture through packing in tub. Butter of varying firmness requires more or less force for proper packing. Where butter is firm there is need of a greater force. This results in increased expulsion of moisture and would explain in part the lower moisture content contained in some of the tubs. On the other hand, it may in part be due to method of sampling. It is not believed that as representative a sample of butter is obtained out of a tub by a trier as is obtained from a composite sample from the churn. In taking a sample there is a certain amount of moisture expressed by trier. This in part may be found on trier and sample and in part may be left in the tub as the trier with sample is being drawn from the butter. The analysis of composite sample from the churn is a method that may be recommended as a safe guide for use of makers. If sample be taken from tubs there is an element of error that is not found in the sampling from churn. To determine the possible variation in different parts of a tub, tubs were stripped and samples taken by a butter trier in a horizontal direction. The first two results represent analysis of samples from trier taken in the usual manner from top downwards. In these two cases about one-third of sample taken by trier was taken to represent that particular part of tub. The following table shows the analysis of samples:

No.	Top	Middle	Bottom
1	13.86	12.20	14.88
2	13.56	12.34	14.00
3	12.98	13.43	13.31
4	13.78	13.35	13.70
5	13.13	12.80	13.21
6	15.61	15.38	15.02
7	13.12	13.42	13.17
8	13.47	13.32	13.58
9	13.17	13.42	13.39
10	15.54		15.57

The variations noticed are probably due more to difficulties in obtaining a representative sample by means of a trier rather than in any real variation present in the tubs. Where the samples are taken horizontally, they would be more fairly representative of that particular portion of the tub. When taken in a perpendicular direction, variation in size of sample taken are noted, the sampler being larger at the upper

end and gradually lessening in size towards the lower end would account in part for variation as shown in No. 1 and No. 2. Then too, the free moisture expressed by trier in cutting the sample is usually found on extracting the sample to be more abundant on the lower portion. This may account for the variation in these two samples. There does not seem to be any reason, other than those explained in connection with variations in moisture content of butter from different parts of the churn, why there should be any variation in tub. This is in part shown by the last eight numbers of the above table.

Where the butter is made in a careless manner and water is held in pockets or as free brine greater variation is possible. To draw general conclusions from above table would require a much more complete and extended set of analyses.

In all the above work many points have been noted. There are a few which require special emphasis. Most of these are already known to the chemist and experiment station worker and they need receive no attention at their hands. To the manager and maker, who has not received training in butter analysis, we commend the following points for consideration:

1. The imperative necessity of correct sampling and preparation of sample.
2. The purchasing of an accurate, reliable scale or balance.
3. The necessity of a separate room for the testing of cream and analysis of butter.
4. Avoidance of draughts in the use of uncovered scales.
5. Control of heating temperature in the evaporation of moisture from butter.
6. Simplicity of method does not meet the needs of incompetent men.